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Development of Flexible Containers for Irradiated Foods

I. Screening of Commercially Available Plastic Laminates

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SUMMARY

In continuation of the effort to find adequate flexible materials for packaging irradiated foods, five commercially available plastic laminates were screened for in-package radiation sterilization processing of bacon, ham and pork. One plastic laminate was transparent. The food-contacting materials were: medium density polyethylene, high density polyethylene, and poly-(vinyl chloride). All samples were packed under vacuum.

Control samples were non-irradiated meat products in flexible pouches (stored at −29°C). The test samples were the meat products packaged in flexible pouches, irradiated at 4.5–5.6 Mrad, Cobalt-60 radiation, and stored at 23°C and 50% R. H. for a 12-month period.

Evaluation phase of the investigation included observations for odor, leakage, and color changes and determination of the physical changes in flexible pouches—heat seal strength, burst strength, and bond strength. Each meat product was subjected to organoleptic and chemical testing. At the end of six months, four of the flexible packaging materials were removed from the storage study because of off-odor and/or off-color that developed in the meat products. Swelling of these pouches was not observed; however, pinholes, delamination and loss of vacuum were noted in the pouches.

One laminate, employing medium density polyethylene as the food-contacting material, was found to be satisfactory for packaging the three meat products and their storage over the one-year period. Changes in physical properties of pouches did not affect their functional performance. Preference scores, obtained from an organoleptic evaluation, indicated that the products were acceptable. Storage time had no pronounced effect on organoleptic acceptability.

INTRODUCTION

The main objective of the Packaging Task of the U. S. Army Radiation Preservation of Foods Program is to develop flexible light-weight containers, capable of withstanding rough handling and storage, which retain protective qualities after storage without any adverse effects on the food. Flexible laminate containers with aluminum foil as an oxygen barrier showed promise in fulfilling the severe requirements (Tripp, 1962).

When the Radiation Laboratory at U. S. Army Natick Laboratories became operational in November 1962, packaging research was directed toward:

(a) determination of the extractives and other fragmentation compounds of various food packaging polymeric materials resulting from ionizing radiation; and

(b) development of flexible containers with chemical, physical and protective characteristics to meet the overall requirements (Payne and Spiegel, 1964; Payne and Long, 1965).

The extractive data were used in a petition submitted by the Army to the Food and Drug Administration to permit the use of six polymeric materials as a food-contacting film in flexible containers for prepackaged foods, gamma irradiated to 6.0 megarad (Adams, 1965). In support of the above research on the development of flexible containers, a food pack study was conducted at Natick Laboratories by screening five commercially available plastic laminates for in-package radiation sterilization processing of bacon, ham and pork. Extractives data obtained from these laminates indicated that six megarad of gamma radiation caused no significant change in extractability of these materials (Garlock, 1964; Wierbicki and Killoran, 1966).

EXPERIMENTAL METHODS

Packaging. The packaging materials used in this study are listed in Table 1. Prior to fabrication of pouches optimum heat sealing conditions (temperature, pressure, dwell time) were established from heat seal data obtained with a Sentinel Model 24A heat sealing machine. Filled pouches were hermetically sealed on a Flex-Vac 6-9 machine, the gauge reading being set at 28 in. vacuum Hg.

Meat Products. Boneless rolled smoked ham was cut into slices with a diameter of 5 in. and a thickness of ¼ in. Each pouch (8 in. × 7 in.) contained 2 slices weighing about 150 g. Boneless pork loins (U.S. No.

Table 1. Packaging materials evaluated.

A	Fabron Inc: 0.5 mil 50 A Mylar/0.5 mil Al foil/2 mil poly(vinyl chloride).[a] (Union Carbide VBA-1144).
B	3M: 30 lb paper/0.35 mil Al foil/0.5 mil polyethylene/0.35 mil Al foil/2 mil Scotchpak 20A5.[a]
C	Phillips: 30 lb paper/0.35 mil Al foil/1 mil Marlex TR-515.[a]
D	3M: 0.3 mil Al foil/30 lb paper/2 mil Scotchpak 20A5.[a]
E	3M: 4.5 mil Transparent Scotchpak 45A27.[a]

[a] Food contacting film.

1) were trimmed, enzyme inactivated by steam heating to center temperature of 74°C, cooled quickly to 5°C, and packaged. Each pouch (11 in. × 7 in.) contained 6 pork loin slices, each ¼ in. thick, weighing about 175 g. Premium quality regular sliced bacon weighing 1 lb was packaged in 11 in. × 8 in. pouches.

Radiation. Cobalt-60 radiation was performed at the Food Radiation Laboratory, U.S. Army Natick Laboratories. Prior to radiation, the prepackaged ham, bacon and pork loin test samples were stored at refrigeration temperature (3°C). Radiation was performed at ambient room temperature. Increase in temperature during radiation was about 20°C. Dose rate was 5.3×10^4 rads per min. Radiation procedure and dosimetry methods were reported previously by Jarrett (1965).

Storage. Irradiated pouches were stored at 23°C and 50% R.H. Nonirradiated frozen control containers were stored at −29°C. Withdrawals for testing both packaging material and food were made at the end of 10, 40, 100, 190, 260 and 365 days.

Packaging Evaluation. At each withdrawal period evaluation of pouches included:

(a) inspection for swells, pinholes and delamination;
(b) leakage test by submerging pouch and maintaining a vacuum of 25 in. Hg for 20 sec for detection of gas bubbles;
(c) free space or theoretical vacuum by the method of Pratt and Kneeland (1965);
(d) oxygen content of headspace gas with a Fisher Partitioner;
(e) seal strength of side seals (machine direction of stock); and
(f) bond strength in the body area between the foil and the food-contacting film.

Both seal and bond strengths were performed by the method described by Payne and Long (1965). A limited number of infrared spectra were recorded with a Beckman IR-9 grating spectrophotometer for irradiated and frozen control food-contacting films from Pouch B. Complete spectra were recorded for the interpretation of the functional group changes in the solid phase using the multiple attenuated total reflectance technique (Hermann, 1965).

FOOD EVALUATION

Toxicity Test. Mouse toxicity tests for detection of *Clostridium botulinum* toxin in irradiated samples were performed immediately prior to organoleptic testing (Whitehair *et al.*, 1964). Total plate counts were determined concurrently with the toxicity tests.

Organoleptic Test. The three meat products were tested by an eight member expert technical panel. Samples were scored for intensity of discoloration (ham), off-odor, off-flavor, mushiness (ham and pork), friability (ham), and radiation flavor on a 9-point scale (1—None, to 9—Extreme). In addition, panelists scored the meat products for preference on the 9-point hedonic scale (Peryam and Pilgrim, 1957). Ham and pork were served without heating. Bacon was oven-fried and served warm.

Chemical Analyses. Both irradiated and frozen control samples were subjected to chemical analyses for pH, thiobarbituric acid test (TBA), peroxide value, and free fatty acid. (TBA values are mg. of malonaldehyde of sample; peroxide values are milliequivalents of peroxide per kg of extracted fat; free fatty acid values are percent as oleic acid.) Sample preparation and analytical procedures were described by Whitehair *et al.* (1964).

RESULTS AND DISCUSSION

For convenience in discussing the results, the aspects of investigative interest can be divided into packaging evaluation, headspace gas data, infrared spectrophotometric analysis, and chemical and organoleptic evaluation of the foods.

Packaging Evaluation. *Pouch B* (Table 1) with medium density polyethylene as the food-contacting film was found to be satisfactory for packaging the three meat products and their storage over the one-year period. At the end of 6 months, irradiated pouches A, C, D and E (Table 1) were removed from the pack test because of off-odor and/or off-flavor that developed in the three meat products. Neither swelling nor leakage was observed in these pouches. However, loss of vacuum and delamination were noted. Specifically, the principal defect in each of the pouches tested was:

Pouch A—adhesive strength between foil and food contacting film was destroyed by the radiation to cause delamination.

Pouch C—a very weak pouch because of thin film in contact with food.

Pouch D—position of paper in pouch made it very susceptible to oxygen wicking into package around its perimeter.

Pouch E—During the first 30 days' storage the three meat products were highly discolored; a response attributable to the transparent pouch.

Table 2 shows that radiation caused a reduction in seal strengths of all pouches tested. For example, seal strength of irradiated pouch B was 30% less than that of frozen control. Table 3 indicates that over the one-year storage period seal strengths of both irradiated and control pouches B with the fatty meat product (bacon) decreased by almost 50%. Table 4 shows that seal strength of pouch A with bacon decreased about 1% over the one-year storage period. Decrease in seal strength was also found with ham and pork, though to a lesser extent, in pouch B. The relatively low seal strength of pouch C with high density polyethylene film in contact with the food was attributed to the thin gauge film (1 mil).

Bond strength and Mullen burst strength data for pouches A, B, C and D are presented in Table 5. Bond strength of pouch A with poly(vinyl chloride) in contact with the foil decreased about 60% after irradiation. This severe reduction is attributed to the loss of adhesion in the bonding agent as a result of radiation. Radiation had but a slight effect on the bond strengths of pouches B, C and D. The relatively high bond strength of pouch B is noteworthy.

Radiation caused about 20% increase in burst strength of pouch A, a 20% decrease in pouches B and D, and a 40% decrease in pouch C. The percentage of decrease in pouch C is not considered reliable because of the inherent weakness attributed to the

1 mil thickness of food contacting film in this pouch.

Headspace Gas and Oxygen Content. The non-destructive test method of weighing pouches under water may be used to calculate free space or the volume of headspace gas after radiation and storage. Thus, a comparison of the volume of headspace gas in irradiated and frozen control samples is a measure of the volume of irradiation induced gases. Typical data on volume of residual gas and oxygen content of irradiated and frozen control meat samples in pouch B are shown in Table 6.

Total headspace gas decreased and oxygen content increased during storage.

Pratt and Kneeland (1965) identified hydrogen as the principal gas produced in foods as a result of radiation processing; other gases, such as CO, CO_2, and CH_4, were present in small amounts. In the same study they reported that in an irradiated foil laminated type pouch containing ham, the hydrogen content of the headspace gas decreased from 47% to less than 1% during storage; on the other hand, in an irradiated tin-plated container with ham, the hydrogen content of the headspace gas did not change during storage. It was conjectured that hydrogen was lost through the seal area along the perimeter of the pouch and to a lesser extent through pinholes in the foil. Increase in oxygen content was attributed to oxygen transmission into pouch from surroundings or "increased percentage" of headspace gas brought about by preferential loss of hydrogen.

Infrared Spectroscopic Examination. Since changes in physical properties of irradiated packaging films reflect radiation-induced chemical changes in molecular structure, an investigation was performed by infrared spectroscopic analysis of subject films before and after radiation. Fig. 1 shows the infrared spectra of the medium density polyethylene (pouch B) as recorded on a Beckman IR-9 spectrophotometer modified for multi-attenuated total reflectance measurements.

Significant changes were noted in the type and distribution of unsaturated groups. The presence of oxygen during radiation had a marked influence on the structural rearrangements. Strongly absorbing trans-type unsaturation (CH = CH) bands at 964 cm^{-1} appear in the spectra after radiation. Vinylidene decay on irradiation is shown by a decrease in the

Table 2. Seal strength [a] of pouches containing ham—irradiated versus frozen control.

Pouch type	Radiation dose (megarad)	Seal strength, g/lineal in. width: 10 days	40 days	100 days	190 days	365 days
A	None	4580	4520	4510	4590	4500
A	4.5	3650	3690	3640	3720	....
B	None	3210	3240	3175	3120	2830
B	4.5	2290	2275	2283	2255	2190
C	None	2250	2225	2200	2030	1910
C	4.5	1870	1920	1850	1830	....
D	None	3275	3300	3290	3200	2900
D	4.5	2476	2500	2520	2430	....
E	None	8500	8560	8440	8360	8210
E	4.5	7360	7350	7290	7270	....

[a] Ten specimens tested; standard deviation: ±180.

Table 3. Seal strength of "Pouch-B" after storage.[a]

Contents	Radiation dose (megarad)	Seal strength [b, c] g/lineal in. width: 10 days	40 days	100 days	190 days	260 days	365 days
Bacon	None	3180	3175	2740	2460	2100	1700
Bacon	4.5	2270	2290	2040	1845	1445	1142
Ham	None	3210	3240	3175	3120	3010	2830
Ham	4.5	2290	2275	2283	2255	2235	2190
Pork	None	3190	3210	3180	3175	3140	3004
Pork	4.5	2285	2320	2290	2240	2150	2165

[a] Irradiated samples stored at 23°C; 50% R.H.
[b] Tested in transverse direction; sealing conditions: Temp 275°C, pressure 40 psi, dwell time 1 sec.
[c] Ten specimens tested; standard deviation: ±180.

Table 4. Seal strength of "Pouch-A" after storage.[a]

Contents	Radiation dose Megarad	Seal strength [b, c] g/lineal in. width: 10 days	40 days	100 days	190 days	365 days
Bacon	None	4540	4560	4490	4530	4484
Bacon	4.5	3680	3700	3710	3675	....
Ham	None	4580	4520	4510	4590	4500
Ham	4.5	3650	3690	3640	3720	....
Pork	None	4520	4510	4560	4580	4490
Pork	4.5	3700	3650	3740	3680	....

[a] Irradiated samples stored at 23°C, 50% R.H.; frozen control at −29°C.
[b] Tested in transverse direction; sealing conditions: Temp 216°C, pressure 40 psi, dwell 1 sec.
[c] Ten specimens tested; standard deviation: ±180.

Table 5. Effect of radiation on bond strength[a] and burst strength.

Pouch	Radiation dose (megarad)	Bond strength [a]: 10 days	Bond strength [a]: 190 days	Mullen burst strength,[b] psi: 10 days	Mullen burst strength,[b] psi: 190 days
A	0	420	390	72	69
A	4.5	160	145	90	81
B	0	720	680	72	65
B	4.5	750	705	55	58
C[c]	0	200	170	27	22
C[c]	4.5	190	165	15	10
D	0	230	200	75	64
D	4.5	210	195	48	54

[a] g/lineal in. width; 10 specimens, standard deviation of mean: ±22.
[b] 10 specimens, standard deviation: ±6.
[c] Tore during test.

Table 6. Pouch B—volume headspace gas and oxygen content during storage.

Meat product	Storage period (days)	Headspace gas,[a] ml/100g: Control	Headspace gas,[a] ml/100g: Irradiated	Oxygen content [b]: Irradiated pouch
Bacon	10	0.3	7.2	0.35
Bacon	190	0.3	5.3	1.13
Bacon	365	0.3	4.5	1.66
Ham	10	0.3	8.1	0.56
Ham	190	0.3	6.1	1.32
Ham	365	0.3	5.2	1.75
Pork	10	0.3	8.2	0.44
Pork	190	0.3	6.3	1.43
Pork	365	0.3	5.4	1.80

[a] Average of five pouches; non-destructive method of weighing under water.
[b] Average of four pouches.

Table 7. Organoleptic intensity values [a] for bacon, ham, and pork after storage.[b]

Food product	Storage time (days)	Discoloration [c]		Off-odor [c]		Mushiness [c]		Friability [c]		Radiation flavor [c]	
Bacon	10	......		1.1	(1.0)	......		......		1.0	(1.3)
	40	......		1.8	(1.2)	......		......		1.4	(1.2)
	100	......		1.2	(1.1)	......		......		1.0	(....)
	190	......		1.3	(1.1)	......		......		1.4	(....)
	260	......		1.4	(1.2)	......		......		1.1	(....)
	365	......		1.3	(1.2)	......		......		1.3	(1.0)
Av				1.35	(1.13)					1.20	(1.17)
Ham	10	2.9	(1.2)	3.3	(1.2)	1.9	(1.3)	......		3.0	(1.2)
	40	3.1	(1.1)	2.3	(1.5)	2.3	(2.0)	......		2.9	(1.6)
	100	2.3	(2.0)	1.7	(1.5)	1.6	(1.8)	......		2.9	(1.0)
	190	......		2.4	(....)	2.0	(....)	2.7	(1.3)	2.6	(1.3)
	260	......		1.9	(....)	2.6	(1.7)	2.9	(1.4)	2.4	(1.0)
	365	2.8	(1.8)	2.1	(1.7)	2.8	(2.0)	2.8	(1.0)	2.8	(1.0)
Av		2.78	(1.53)	2.28	(1.48)	2.20	(1.76)	2.80	(1.23)	2.77	(1.15)
Pork	10	......		2.2	(1.8)	......		......		2.2	(1.3)
	40	1.5	(1.1)	1.6	(2.0)	1.3	(2.0)	......		2.3	(1.2)
	100	1.4	(1.4)	2.9	(1.9)	2.9	(1.5)	......		2.0	(1.0)
	190	......		2.0	(....)	1.8	(1.4)	......		2.2	(....)
	260	1.3	(1.4)	2.1	(1.8)	2.4	(1.3)	......		2.3	(1.6)
	365	1.1	(1.0)	2.1	(1.8)	2.6	(1.4)	......		1.8	(1.6)
Av		1.32	(1.22)	2.15	(1.86)	2.20	(1.52)	......		2.13	(1.34)

[a] Intensity scale used: 1—none, 2—trace, 3—small, 5—moderate, 7—strong, and 9—extreme.
[b] Mean scores; 8 panelists.
[c] Frozen control values are in parentheses.

$R_1R_2C = CH_2$ band at 888 cm^{-1}. There is an increase in both hydroxyl (3350 cm^{-1}), and carbonyl (1730 cm^{-1}) bands, indicating the formation of oxidation products.

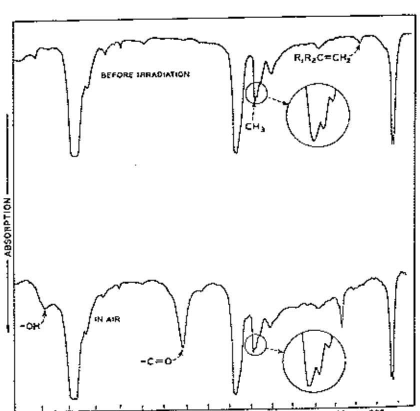

Fig. 1. The effect of Cobalt-60 irradiation (6 Megarad) in air on medium density polyethylene.

Chemical and Organoleptic Food Evaluation. *Toxicity.* No toxicity symptoms of any type were observed in young white mice injected intraperitoneally with the filtrates of the irradiated meat samples after storage up to one year.

Bacteriological. Plate counts of all irradiated samples used in organoleptic testing were less than 10 microorganisms per g—based on the smallest dilution plated. Even though the frozen control samples were stored at −29°C, they contained moderate number of microorganisms ranging from 10^3 to 10^4 gram of sample.

Organoleptic. Table 7 lists the intensity scores for pertinent undesirable characteristics that are present in irradiated meat samples. Table 8 presents the preference scores for the same meat samples. Although the intensity and preference score data were not analyzed statistically, they do permit specific conclusions based on our extensive experience with irradiated foods.

In general, the intensity scores (Table 7) ranged from 1 (none) to 3 (slight), indicating small off-flavor, etc., in the three foods investigated. Even though the panelists were trained to detect small differences in the quality characteristics of the irradiated foods, several panelists gave similar scores to the frozen control samples, indicating that one is dealing with differences that are very small and hardly detectable by an expert panel. Based on the evaluations of the panel the following conclusions can be made for the test samples:

Bacon: Irradiated samples were comparable to the control in that they were free from off-odor and irradiation flavor.

Ham: Slightly less intense cured meat color (discoloration in Table 7) was noticed in irradiated samples. Off-odor and irradiation flavor were also detected, as is always the case for ham samples irradiated at ambient room temperature with a dose of 4.5 megarad. Irradiated samples were more tender than control samples as shown by slightly increased mushiness scores; however, they showed a tendency to fall apart more easily than the control samples as shown by the friability scores.

Pork: As ham, the irradiated pork samples showed a slight increase in off-odor, mushiness, and irradiation flavor compared to the control samples. Although the data for "discoloration" don't show any difference between irradiated and control samples, normally enzyme-inactivated pork irradiated at a dose of 4.5 megarad has a pink color inside the samples. The relatively thin samples used in this study were probably responsible for the absence of pink color.

Preference scores (Table 8) indicated that the three irradiated meat products were acceptable. Bacon and pork samples were rated as equally acceptable as frozen controls. Ham samples were rated lower than frozen controls, probably because of the slight irradiation flavor, mushiness, and discoloration as shown in Table 7. Storage time had no pronounced effect on the preference scores.

Chemical Data. Mean values of duplicate chemical analyses are listed in Table 9. The values obtained for oxidative rancidity as measured by the 2-thiobarbituric acid (TBA) test

Table 8. Preference scores [a] for bacon, ham, and pork-packaging: "Pouch B."

Storage time (days)	Bacon [d]		Ham [d]		Pork [d]	
10	7.3[b]	(7.0)[c]	5.7	(8.3)	5.7	(5.8)
40	....		5.3	(7.1)	6.1	(6.3)
100	7.0	(....)	5.9	(7.1)	5.8	(6.1)
190	7.4	(....)	5.8	(....)	6.9	(....)
260	7.1	(....)	6.5	(....)	6.4	(....)
365	7.5	(7.3)	5.6	(6.3)	6.1	(6.6)
Av	7.26	(7.15)	5.96	(7.20)	6.15	(6.20)

[a] Mean scores for eight member panel; range of scoring: 9, like extremely, 1, dislike extremely.
[b] Irradiated sample mean values.
[c] Frozen control sample (−29°C) mean values.
[d] Bacon served fried; ham served cold; pork served cold.

Table 9. Chemical data [a] for irradiated and frozen control samples: "Pouch B."

Meat product	pH		TBA [b]		Peroxide [c]		Free fatty acid [d]	
	Initial	1 yr.	Initial	1 yr.	Initial	1 yr.	Initial	1 yr.
Bacon								
Control	6.74	6.12	0.10	0.74	0.22	1.2	1.31	2.7
Irradiated	6.60	6.31	0.16	0.18	0.12	0.61	1.28	1.7
Ham								
Control	6.30	6.23	0.13	0.26	11.7	13.2	......	0.88
Irradiated	6.28	6.41	0.21	0.08	4.5	8.8	......	1.50
Pork								
Control	5.80	6.13	7.5	11.4	5.2	63.0	......	1.1
Irradiated	5.81	6.04	1.6	0.99	1.6	5.2	......	1.7

[a] Average of duplicate analyses.
[b] Mg. malonaldehyde per 1000 g. of sample.
[c] Milliequivalents of peroxide per 1000 g. extracted fat.
[d] Percent as oleic acid.

appear to be low for both frozen and irradiated bacon and ham samples. Saslaw (1965) presented data indicating that the TBA test might not be totally reliable as a measure of the extent of oxidation of the naturally occurring highly unsaturated fatty acids in irradiated foods. It was found that besides malonaldehyde other "unidentified carbonyl compounds" react with TBA.

The TBA data for pork indicate that irradiated samples were less oxidized than the frozen control samples. Peroxide values for irradiated products are lower than those of frozen control samples. This reduction could be attributed to (1) less available oxygen being present (in vacuum packaging) to react with free radicals produced by irradiation, and (2) accelerated decomposition of fat hydroperoxides normally present in the meat products. Differences in free fatty acid values of irradiated and frozen control samples are very small. The higher values for the irradiated ham and pork samples may be attributed to direct action of radiation.

CONCLUSIONS

Four of the five commercially available flexible materials evaluated did not have the required protective qualities and resistance to radiation for use in prepackaged radiation sterilized meat products.

One laminate, employing medium density polyethylene as the food contacting film, was found to be satisfactory for packaging bacon, ham and pork and their storage over the one year period. Preference scores, obtained from the organoleptic evaluation, indicated the products were acceptable. Storage time had no pronounced effect on organoleptic acceptability.

Research in the development of flexible lightweight containers for irradiated foods is being continued both in-house and on contract. Areas of study include (a) determination of the effects of prolonged storage and rough handling on protective characteristics; (b) improved permeability resistance to oxygen intake during storage; and (c) evaluation of candidate films that will perform satisfactorily in laminates for prepackaged electron irradiated foods—radiation being carried out at −65°C to −80°C.

It should be noted that flexible pouches used in this study must be enclosed in a protective jacket to form a functional package.

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